Quicksilver and Quick-thinking: Insight into the Alchemy of Mercury
A new interdisciplinary research to discover the chemical reality of ancient alchemical recipes

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Abstract
Ancient alchemical recipes represent an invaluable source to understand how our ancestors described and conceptualised nature. Such recipes were often dismissed as nonsense, or even interpreted as mere allegories for spiritual practices of self-purification. Our research adopts a totally new interdisciplinary outlook through collaboration between chemists and historians of science, to investigate one of the most intriguing of elements, that is mercury. A corpus of ancient sources —in Greek, Latin and Syriac—was scoured in search of information on the extraction of mercury from cinnabar. Following the sources, we replicated the recipes and explored the viability and feasibility of the reactions, as well as the technical underpinnings of the texts, in order to disclose the practical dimensions of ancient alchemy and an unexpected variety of reactions.

Introduction
Ancient alchemical procedures have been transmitted over centuries as written recipes that scribes copied onto manuscripts hundreds of times. A careful philological analysis is essential to disentangle this rich textual tradition and translate ancient recipes into modern renderings. However, written instructions that deal with practices may remain obscure without their words being turned into deeds: only by re-enacting these practices in modern laboratories it can be possible to anchor such ancient texts to the chemical reality that they were intended to encapsulate.

In this study, we propose a suite of philologically-informed experiments that apply, for the first time, a circular hermeneutical approach to ancient alchemy: we read the sources, propose chemical interpretations, test these hypotheses in the laboratory and return to the sources. First, we followed the instructions of ancient recipes and used synthetic reagents, which enabled us to characterise the products and detect the roles played by the different ingredients. Then, we reproduced the recipes using mineral ores to confirm the possibility of the reaction with substances similar to those that were used by the ancients. In terms of equipment, we did not opt for historical reconstructions: without affecting the results of the tested reactions, we adapted modern labware, thus significantly reducing the experiments' durations and conforming to modern safety protocols.

Following this transdisciplinary approach, we explore the ancient chemistry of mercury, the natural element that held the greatest fascination among alchemists. In particular, we investigate Greek, Latin and Syriac texts that describe its cold and hot extraction processes from cinnabar. This study rectifies some explanations that have been commonly proposed for ancient alchemical knowhow and leads us to a better understanding of how ancient practitioners conceptualised and processed mercury. In following these ancient procedures, we were even able to identify reactions that never found their way into modern chemistry laboratories and the literature.

Cold Extraction

The earliest known procedure for extracting mercury is recorded in Theophrastus' *On stones* (fourth century BCE), which states that[1]

'Mercury is produced by grinding cinnabar with vinegar in a copper mortar with a copper pestle'.

In the 1920s, the chemist and historian of science, Kenneth C. Bailey, made the first and only attempt to replicate this procedure by grinding cinnabar and copper turnings with vinegar within a mortar.[2] The cinnabar turned black – a chromatic change that indicates the presence of copper sulphide, whereas, the surface of the copper turnings became covered in a copper–mercury amalgam, which also featured in our replication (see Figures S1 and S2 in the Supplementary Information [SI hereinafter]).

After Bailey's work, no further experiments were carried out and the procedure fell into oblivion until Takacs cited Theophrastus' recipe as being the earliest witness to a mechanochemical reaction.[3]

Modern chemists immediately recognise copper—i.e. the metal from which pestle and mortar are made—as being a key reagent. However, this procedural leap from instruments to reagents is not self-evident when observed from a historical perspective. Theophrastus' method was again reported by Pliny the Elder[4] and the Graeco–Egyptian alchemist Zosimus of Panopolis (third–fourth century CE), who introduced interesting variations.[5,6,7] Zosimus claimed that Maria the Jewess, designer of the famous *bain-marie*, and Chymes, the eponymous hero of (al)chemy, used to grind cinnabar with pestles and mortars comprised of various metals, such as lead and tin. These sources may have conveyed the idea that metals constituted a key ingredient. Indeed, in a recipe only preserved in Syriac translation, Zosimus prescribes the grinding of cinnabar with copper filings and vinegar until droplets of mercury become visible.[8] The metal is thereby added to the procedure as an independent reagent.

We initially tested this reaction by manually grinding synthetic cinnabar (HgS) and copper (Cu in powder) together with some drops of glacial acetic acid. The powder slowly turned black and after one hour of grinding, droplets of mercury (Hg), never recorded in any previous experimental report, were clearly visible (see Figure 1).
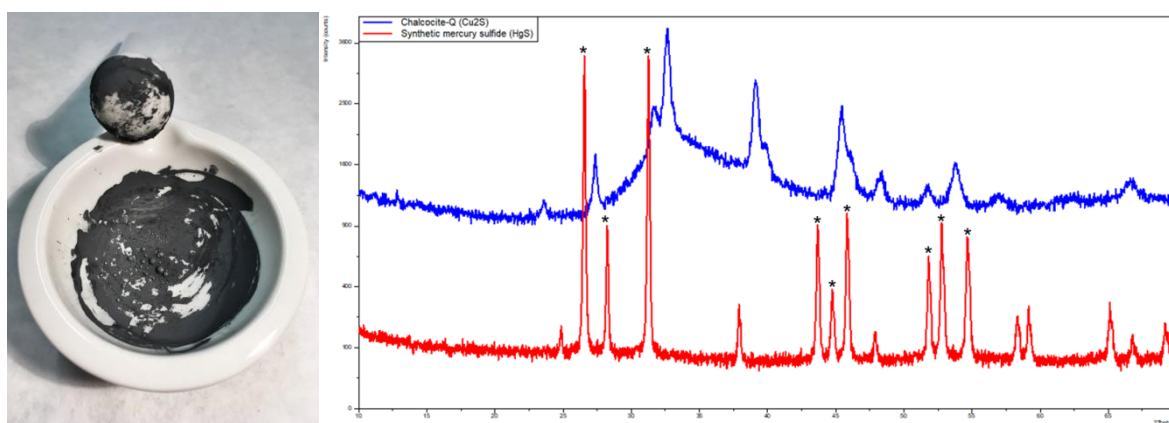

Figure 1. Left: ceramic mortar in which HgS and copper powder were ground—droplets of mercury are visible at the bottom. Right: comparison between XRPD pattern (blue solid line) of the residual powder obtained from the reaction of synthetic mercury sulfide and copper powder in the presence of acetic acid (reaction mixture ground with a ball mill for four hours at 25 Hz; the high background in the pattern is due to the presence of amorphous phase of metallic mercury in the final powder) and XRPD pattern of synthetic mercury sulfide (red solid line). The asterisks in the red XRPD diffractogram indicate the peaks assigned to the cinnabar. Phase identification was performed using the *PDF 2 Release 2004* database.

By ball milling cinnabar and copper together with some drops of glacial acetic acid, after six hours the reaction was complete. The X-ray powder diffraction pattern (XRPD) is characteristic of Cu_2S Chalcocite-Q (Figure S4), meaning that the reaction comprised HgS + 2Cu→ **Cu_2S** + Hg, which is unexpected if compared with what has been reported in recent studies. If Takacs' article identifies the product of the reaction with mercury and generic copper sulphide, when this study began to be cited, other researchers limited the identification of different possible sulphides to 'probably CuS', without conducting further analyses;[9,10,11]; eventually, in recent papers, the reaction was formalised as: HgS + Cu → **CuS** + Hg. Our experiments make it clear that Chalcocite-Q (**Cu_2S**) is the reaction product and not the incorrectly-reported Covellite (**CuS**).[12] This suggests that the reaction has only been cited in the scientific literature, without being reproduced and characterised. In fact, as in the case of the transmission of ancient recipes, which have been repeatedly copied in various manuscripts, modern explanations, when cited from one scientific paper to another, can be misinterpreted and their inaccuracies amplified over time.

All ancient sources stress the key role played by vinegar or water in the procedures. In our experiments, the mechanochemical reaction with copper and cinnabar was performed with glacial acetic acid, 6% acetic acid (as in vinegar), water or in dry conditions. Droplets of mercury always became visible, but it is clear that the acid plays a catalytic role, perhaps by removing passivation on the metal surface.

With respect to the use of metals other than copper, the replications confirmed that lead (Pb) can reduce mercury: the XRPD pattern (see Figure S5) indicates the presence of lead sulphide, but no droplets of mercury are visible owing to the formation of a powdery HgPb amalgam. When the reaction occurs with tin (Sn), the formation of the amalgam (HgSn) is clearly obtained, but the product of the oxidation of tin, probably tin sulphide, is hardly observed in the XRPD (for more details, see the SI). This amalgam is a soft silvery paste (Figure S7), which ancient alchemists may have easily identified as being a kind of quicksilver: indeed, in their writings, they classified different types of mercury in accordance with the metals used in its extraction.[13]

Hot extraction

According to ancient sources, the hot extraction method of processing mercury from cinnabar can be divided into three procedures: 1) simple heating of cinnabar; 2) heating cinnabar in a closed vessel in the presence of iron; and 3) heating cinnabar in the presence of so-called *nitron oil*. Up until now, this variety has escaped the attention of modern historians, who simply refer to the well-known reaction of cinnabar in the presence of oxygen at high temperatures: $HgS + O_2$ → SO_2 + Hg.[14]

This procedure, which primarily entails the roasting of mercury ores, became standard after the publication of Agricola's *De Re Metallica* (1556) onwards, whereas classical sources are less precise about the process. The Roman architect Vitruvius (first century BCE) only records an extempory observation, noting that to produce the pigment, the ancients used to dry moist clods of cinnabar in ovens. When the fumes condensed, mercury could be found at the bottom of the oven and on the clods.[15] If the ores encased droplets of mercury, it would have simply been distilled out when the ores would have been heated.

This procedure is not described in other ancient recipes, which instead specify the use of closed vessels to facilitate the recovery of mercury and protect ancient alchemists from its fumes, whose toxicity was already well-known in antiquity.[16] However, in a closed environment the procedure was probably less efficient. We tested this by heating the cinnabar ores within an alumina crucible that was covered with a lid. In a crucible, the

cinnabar sublimates and partially reacts with atmospheric oxygen, yielding mercury that condenses on the lid. The amount of oxygen estimated to be inside the crucible is insufficient to complete the reaction. Indeed, residual cinnabar and black metacinnabar (polymorphically-stable at high temperatures)[17] were always observed, also after prolonged heating.

The second method entails a dramatic shift in the choice of ingredients, as described by Pliny the Elder,[18] Dioscorides and, a few centuries later, by the alchemist Christianus.[19] Dioscorides (first century CE), for instance, wrote:

'They put an iron shell containing cinnabar in an earthenware casserole and enclose it with a convex lid smeared on with clay; then they light a fire upon the casserole with charcoal. They wipe off the vapour settled on the lid, that is quicksilver'.[20]

We replicated this procedure by placing the cinnabar on a small iron (Fe) plate within an alumina crucible that was then covered with a lid (see Figure 2). Upon heating, cinnabar reacts with iron – and not with oxygen – according to the following reaction: $HgS + Fe \rightarrow FeS + Hg$.[14]

The reaction swiftly reaches completeness and the residual powder is characterised by XRPD, which confirms the presence of iron sulphide (FeS; figures S13 and S15). Despite the crucial role played by iron as a reagent, this has been overlooked in the modern studies of ancient extraction technologies.[21]
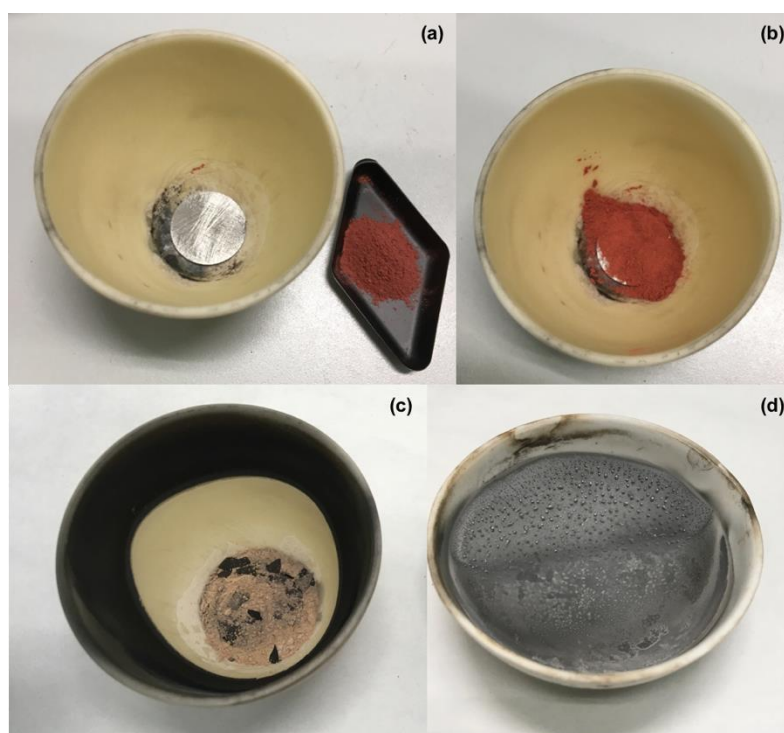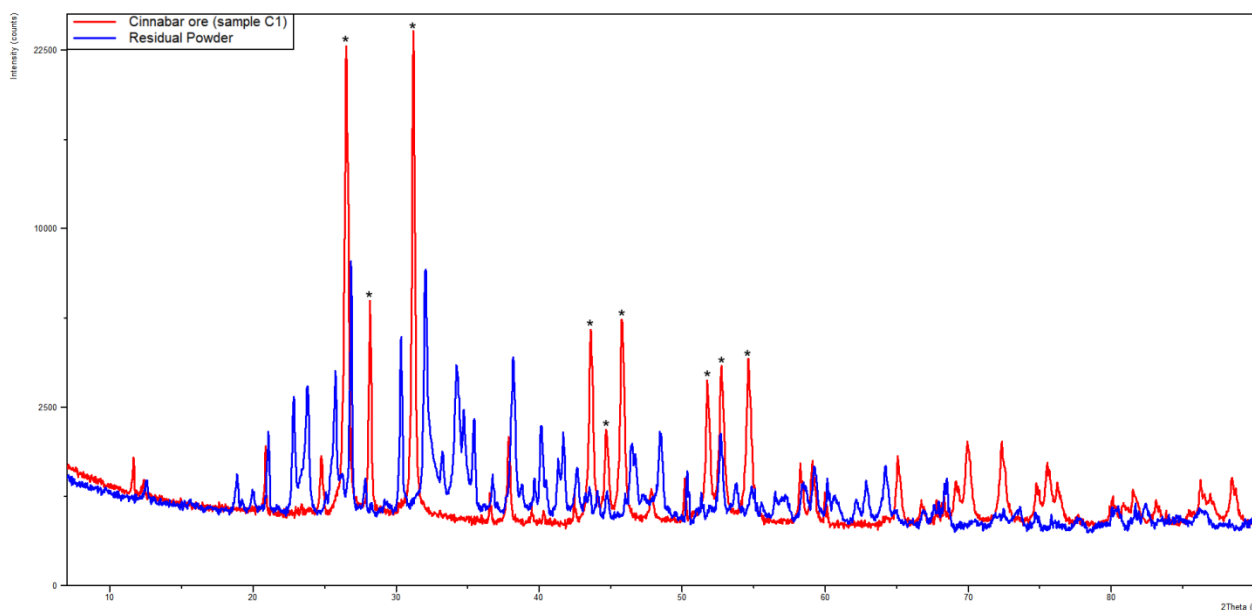
Figure 2. Top: reaction vessel with iron plate and cinnabar powder prior to the reaction (Images (a) and (b)) and after 10 min of heating using a Bunsen burner (Image (c))—the mercury condensed on the lid (Image (d)). Bottom: Comparison between the XRPD of the cinnabar ore (sample C4, red solid line) and the residual powder obtained from the reaction of the cinnabar ore (sample C4) and iron plate (blue solid line); the reaction mixture was heated with a Bunsen burner. The asterisks in the red XRPD diffractogram indicate the peaks assigned to the cinnabar.

The third procedure was described by the alchemist Pseudo-Democritus (first century CE), as reported by a late Byzantine author: Democritus is said to have ground cinnabar together with *nitron oil* and placed the mixture in a 'double vessel' in order to capture all of the vapour; that is, the mercury lying idle in the cinnabar.[22] Byzantine manuscripts also transmit the recipe:

'You must know that the transformation of cinnabar happens by means of *nitron oil*: in this way cinnabar is melted by a light fire, as you know'.[23]

What is in fact meant by 'nitron oil' is elusive and remains a matter of debate. *Nitron* probably refers to the mineral natron (sodium carbonate decahydrate) that was extracted from an ancient mining site known today as *Wadi el-Natrun* – a nearly dry lake in Egypt. However, its association with 'oil' is uncertain. The alchemists probably referred to a mixture prepared by dispersing natron in a solvent. After all, they believed that liquid substances could react more promptly than solids, as had already been asserted by Aristotle.[24] If we consider the compounds that were known at the time, this mixture likely constituted a solution of sodium carbonate dissolved in water, oil (*e.g.*, castor and linseed oil), or vinegar. This supposition is supported by a Byzantine alchemist, who identified *nitron oil* as the 'vinegar of *nitron*'.[25]

In a closed vessel, the role of sodium carbonate is pivotal: we mildly heated the same amount of cinnabar with and without sodium carbonate (Na_2CO_3) by not adding any liquid. In the former case, the cinnabar fully converted into mercury; in the latter, unreacted cinnabar remained (see Figure 3).
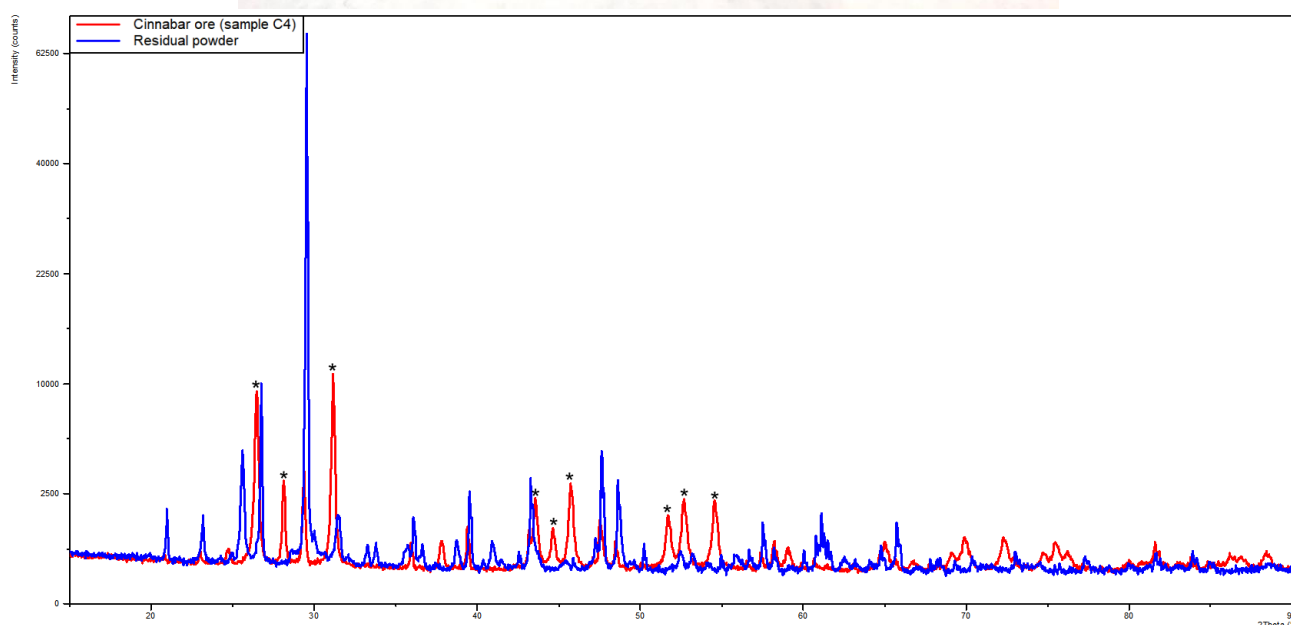
Figure 3. Top: residual powders obtained after 48-h heating at 300°C—a mixture of cinnabar ores and Na_2CO_3 (left) and the cinnabar ore (right). Bottom: comparison between the XRPD of the cinnabar ore (sample C1, red solid line) and the residual powder obtained from the reaction of the cinnabar ore (sample C1) with sodium carbonate (blue solid line); the reaction mixture was then heated with a mantle (at 300 °C) for 48 hours. The asterisks in the red XRPD diffractogram represent the peaks assigned to the cinnabar.

To confirm the role played by sodium carbonate, we performed the extraction under vacuum to prevent the cinnabar reacting with atmospheric oxygen: under this condition, mercury was obtained and sodium sulphate (Na_2SO_4) identified by an XRPD analysis as the product (see Figure S43).

In the contemporary literature, this reaction was only reported once in a fairly old paper by Berthier,[26] in which the residual powder is described as a mixture of sodium sulphate (Na_2SO_4) and sodium sulphide (Na_2S). However, in all our experiments, no sodium sulphide was detected using a XRPD or qualitative analysis by means of sodium nitroprusside solution,[27] whereas, sodium sulphate was always observed.

Once the key role of sodium carbonate in the hot extraction of mercury had been confirmed, the nature and role of *nitron oil* remained to be determined. We prepared saturated solutions of sodium carbonate in water or vinegar to mimic the viscosity of oil. It

is well-known that when mixed with vinegar, sodium carbonate reacts forming sodium acetate and carbon dioxide bubbles; this fact was perhaps recorded by a Byzantine alchemist who claimed that *nitron* effervesces when placed in a liquid.[28] However, the low acetic acid concentration in vinegar does not fully consume sodium carbonate.

In order to carry out the extraction, we used sodium carbonate dissolved in water or vinegar: the water (also contained in the vinegar) evaporates, and sodium sulphate and mercury are produced. In both cases, no significant improvement was observed by comparison to the reactions that occurred in dry conditions.

Finally, we tested a mixture of sodium carbonate and vegetal oil and found that even with mild heating, the oil easily burns and its decomposition prevents the extraction of mercury. If the temperature is reduced, the burning is avoided, but the extraction does not proceed.

To summarise, like iron in the second method described, *nitron* serves as a reducing agent in the reaction when carried out within a closed apparatus. The use of such equipment, always emphasised in ancient alchemical texts, contrasts with the common assumption that mercury was produced by simply roasting cinnabar in the presence of oxygen.

Conclusions

A combined philological and experimental investigation of ancient sources was conducted, and it was demonstrated how crucial procedural information can often be found in the simple textual details of ancient recipes. For instance, an iron shell turned out to be no mere sample-holder but enabled a reaction with a fundamental ingredient to take place.

Similarly, in cold extraction, copper was initially provided by the instruments (pestle and mortar) and later developed into a steady addition in the form of copper filings. *Nitron oil* was also added, as it improved the extraction yield. The problematic identification of some substances and their dubious experimental role led us to wonder whether the choices of ingredients were not only based on technical factors but also influenced by cultural aspects. *Nitron*, in particular, had a profound religious value in Graeco-Roman Egypt. The walls of Graeco-Egyptian temples feature recipes for the production of *nitron* balls, which were used to cleanse the metallic statues of gods, as well as to transform corpses into mummies as perfected bodies for the afterlife.[29] This ritual background could have affected the choices of alchemists in their 'experiments', which were intended to purify, transform and enhance certain substances. Cultural considerations, with technical observations, may have served as driving forces in ancient alchemical practices. These elements were at the core of the investigation reported in this study that began by exploring what ancient alchemists did, and evolved into an analysis of how and why they operated as they did.

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